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(54) **Extruding a continuous polymeric encapsulant of varying composition over a metal line**

(57) The metal line is suitable for transmitting signals or injection of chemicals in a hostile subterranean environment. The encapsulant is continuously extruded as sequential sections, comprising a first nylon-based material section and a second fluoropolymer-based material section, and an intermediate section wherein the composition of the encapsulant is a composite of varying percentages of the nylon and fluoropolymer-based materials. The metal line is preheated so that the extruded polymer does not stress crack, and the composite material is extruded at a temperature substantially above the melting point of nylon, and is fluid cooled to delay crystallisation. The encapsulated tubular metal line is suited for transmitting fluid through a petroleum recovery well bore having a corrosive fluid wherein the temperature in the well bore varies with depth. The continuous extrusion between the different sections, avoids the leakage that might otherwise occur, between seals at the section junctions.

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This print takes account of replacement documents submitted after the date of filing to enable the application to comply with the formal requirements of the Patents Rules 1982.

DESCRIPTION

EXTRUDED VARIABLE ENCAPSULATED SUBSURFACE LINE AND METHOD

The present invention relates to elongate lines with extruded encapsulants and, more particularly, to techniques for continously extruding an encapsulant over a metal line suitable for subsurface signal transmission or chemical injection.

Encapsulated lines have long been used for transmitting fluid, fluid pressure or electrical signals through hostile environments to subsurface locations. In the petroleum recovery industry, for example, it is common to provide a metal injection line secured to the exterior of tubing as the tubing is lowered into a well bore. The injection line may be used to transmit from the surface to one or more downhole tools, and may also be used to inject a desired chemical into the formation to enhance recovery of well fluids. To minimise the rupture of the injection line over a period of time due to corrosion and/or abrasion of the downhole fluids, such injection lines have long been covered with a selected polymer, such as nylon. This encapsulating material is typically applied by extruding the nylon on the metal injection line, so that the nylon sufficiently covers the exterior of the metal injection line.

As geologists search deeper into the earth for hydrocarbons, the temperature of the well bore and thus the temperature to which the injection line is subjected generally tend to increase. Moreover, it is frequently known that certain sections of a well bore will be subjected to one type of fluid environment, while another section is subjected to a more hostile environment. Accordingly, one type of

polymer may be ideally suited as the encapsulant for an upper, less corrosive section of the well bore, while another type of polymer is ideally suited as the encapsulant for a lower, higher temperature section of the well bore. One polymer used for "deep well" injection lines is a fluoropolymer, which exhibits high chemical resistance and good barrier properties.

The problem, however, remains with providing a reliable seal between the different material encapsulants for different sections of a subterranean injection line. Since the injection line is typically continuous from the surface to the selected depth in the well bore, a leak along any portion of the injection line practically results in failure of the entire injection line. The cost of repairing the injection line at this point of the leak is negligible compared to the cost of retrieving and reinserting the injection line in the well bore, and the cost of the resultant delay in production of hydrocarbon from the formation. Any seal between different material encapsulants for an injection line must therefore be fluid-tight to prevent well fluids from contacting the metal injection line over a prolonged period of time. Moreover, any seal between these different encapsulants generally cannot have a diameter significantly greater than the diameter of the encapsulated injection line. An expanded diameter section of the encapsulated line will typically be quickly worn as the injection line and the tubing are lowered into the well, and may cause a downhole "hang-up" which in turn causes lost time and expense.

As a result of the disadvantage and problems associated with providing a reliable seal between two different encapsulating polymers on a single injection line, the prior art has long selected the

polymer for an injection line which is best able to withstand the most hostile environment conditions to which any section of the injection line will be subjected. In other words, while a nylon material encapsulant might be ideal for the upper 5,000 feet of a bore hole and a fluoropolymer encapsulant suitable for the lower 5,000 feet of a bore hole, the prior art has typically selected the more heat stable and heat and chemical resistant fluoropolymer as the encapsulant for the entire 10,000 feet of injection line. This "single polymer" selection accordingly results in substantially increased costs for the encapsulated injection line, since a suitable fluoropolymer encapsulant is typically much more expensive than a suitable nylon encapsulant. Moreover, this "single polymer" selection may result in the use of material for the encapsulant of a portion of the injection line which is not well suitable for the environment in which that section of the injection line is positioned.

The disadvantages of the prior art are overcome by the present invention, and an improved variable composition encapsulant and method of forming the encapsulant on a line suitable for subsurface use are hereinafter disclosed.

According to a first aspect of the present invention, a method of encapsulating an elongate metal line suitable for positioning in a subsurface well comprises extruding a first-selected polymer circumferentially about a first section of the line having a preselected length; extruding a second-selected polymer circumferentially about a second section of the line having a predetermined length; and extruding a composite polymer circumferentially about an intermediate section of the line between the first and second sections, the composition of the composite polymer varying along

the intermediate length of the line from a high first-selected polymer/low second-selected polymer composition to a low first-selected polymer/high second-selected polymer composition, such that a continuous extruded encapsulant is formed interconnecting the first- and second-selected polymers, and substantially no part of the extruded mixture having substantially equal proportions of the first- and second-selected polymers.

The encapsulant is continuously extruded about the metal line, whether it be a fluid conduit or an electric conductor to produce no discernible material change interfaces.

Preferably, the first-selected polymer is a nylon-based polymer and the second-selected polymer is a fluoropolymer-based polymer.

Also, the composition of the composite polymer may vary uniformly with the length of the intermediate section.

The metal line is preferably heated before applying the nylon/fluoropolymer material, thereby reducing the likelihood of stress cracks subsequently developing in this extruded section. The temperature of the extruded composite material varies according to a preferred technique wherein the nylon is heated to a temperature above its melting point, but below its decomposition point, and the fluoropolymer is heated to a temperature just slightly in excess of its melting point. The composite material is preferably cooled in the air to delay crystallisation of the composite polymer.

According to a second aspect of the present invention an encapsulated metal line suitable for positioning in a hostile subsurface environment, comprises a first-selected polymer encapsulant circumferentially positioned about a first length of the line; a second-selected polymer encapsulant

circumferentially postioned about a second length of the line; and, a composite polymer circumferentially positioned about an intermediate section of the line between the first and second lengths, the composition of the composite polymer varying along the intermediate length from a high first-selected polymer/low second-selected polymer composition to a low first-selected polymer/high second-selected polymer composition, such that a continuous encapsulant is formed interconnecting the first- and second-selected polymer encapsulants, and substantially no part of the extruded mixture having substantially equal proportions of the first- and second-selected polymer.

A feature of the present invention is that a reliable encapsulant may be applied to protect the entire length of a metal line to be positioned in different environments along its length, such that a selected material for the encapsulant can be based on the differing anticipated conditions along the length of the line, thereby substantially reducing the cost of providing a suitable encapsulated line.

An advantage of the present invention is that the encapsulated line can constitute a metal tubing suitable for use as an injection line in a subterranean well bore. The intermediate section of the encapsulant does not increase the diameter of the injection line so that problems associated with excessive wear of an enlarged diameter encapsulated section and with hang-ups in a well bore are avoided.

These and other objects, features, and advantages of the present invention will become apparent from the following detailed description.

The first selected polymer, which is extruded circumferentially about a line, such as copper, stainless steel or other fluid conducting tubing, or electrical wire, will be nylon-based. Preferably,

such nylon will be modified nylon 6, which is an extrusion grade resin for tubing applications. Typical of such nylon 6 materials, in modified form, is a heat stabilised CAPRON 8254 HS, of Allied Plastics, Morristown, New Jersey. Typically, the selected nylon for use in the present invention will have the typical properties shown in Table 1.

The second selected polymer will be a fluoropolymer based material which is derivative of a fluoropolymer resin. Preferably, the selected fluoropolymer utilised as the second polymer will have key properties approximating or equal to those shown in Table 2.

Such a fluoropolymer resin material is marketed under the trademark HALAR by Ausimont, Morristown, New Jersey. A particular fluoropolymer resin is marketed under the trademark HALAR-ECTFE, having the physical properties shown in Table 3.

In the present invention the first and second polymer materials are extruded into the metal line to form three basic coatings. The first coating, for lower temperature usage, will be made of the first selected polymer, followed by a length of composite polymer which will be a combination of varying percentages of the nylon-based polymer material and a fluoropolymer-based material. The third coating for high temperature exposure will be the fluoropolymer-based material. In preparing the metal line for encapsulation, an extruder system may be utilised. Such a system is well known to those skilled in the art, and comprises a device with a rotating screw turning inside a heated barrel. The screw is rotated by an external drive and the barrel of the extruder is heated by electric heating units, usually with three or more of the variable temperature units located along the barrel. The screw enters one end of the extruder barrel while the opposite end is

fixed with an extrudate head and die. The die can be variable in design, dependent upon the particular diameter and lengths of the lines to be encapsulated in the present process. The head and die area also are heated. At the open end of the barrel, a material hopper is located above the rotating screw to allow introduction of the various types of materials to be extruded.

The temperature ranges for the extrusion of the first and second selected polymers are as shown in Table 4.

In the encapsulation method of the present invention, the extrusion process requires that the actual temperatures be in the upper range for the first selected polymer, above, and in the lower range for the second selected polymer, above. As encapsulation extends from the first selected polymer through the composite polymer, which is a combination of the first and second polymers, to the second polymer, the temperatures in each zone may be adjusted by increasing or decreasing the temperatures, depending upon which polymer is the predominant component in the composite polymer.

In order to obtain optimum properties for encapsulation, the extruded polymer material on the line should be air cooled for such time as the extrudate has solidified to the point of not being deformed or imprinted by light finger pressure, or a minimum of about 60 seconds, whichever is longer. Thereafter, the encapsulated liner may be water and/or air cooled.

Prior to initiation of encapsulation, the selected line should be pre-heated before entering the die head. Such pre-heating should be performed at a temperature of from between about 300°F and about 400°F in order to help ensure that the extrudate does not crystallise too rapidly, thus

causing high stress areas which are subject to fracture after cooling and re-heating.

Prior to performing the encapsulation method, any selected encapsulation materials which may be hygroscopic must be dried. The extruder and extruder barrel are preheated to the processing temperatures desired, as described above. The material to extrude is purged through the barrel to ensure that no contamination of previously processed material occurs. The line is then loaded in the delivery system and assembled into the die. The extruder screw is rotated and the extrudate is allowed to fill the screw, barrel and die head until a consistent melt is achieved. A take-up mechanism is started to pull the line through the die. The screw rpm rotation is increased to create and maintain the desired dimensions and configuration of the extruded product. Thereafter, the material is cooled and the extruded product is wound on a take-up reel, or other device, for storage purposes.

In order to determine the effects of increase in volume, if any, and hardness variations of lines encapsulated using the present process, stainless steel lines were coated with a nylon-based polymer as the first selected polymer and a HALAR material as the fluoropolymer-based composition of the second selected polymer, with a section of the encapsulated line forming a composite of each of these polymers in varying percentages. The length of encapsulated line was exposed for 160 hours at 300°F in No. 2 diesel fuel containing an acid corrosion inhibitor. Another test was run for 504 hours, 250°F with an acid corrosion inhibitor. The samples were collected from regions of the encapsulated line marked as 100% first selected polymer; 90% first selected polymer/10% second selected polymer; 50% first selected polymer; 50% second selected polymer; 10% first selected

polymer/90% second selected polymer; and 100% second selected polymer. Three samples from each region were cut with lengths being between 1-1/2 to 2-1/2 inches. All samples were weighed, measured for hardness, placed on a fixture and immersed in the diesel/inhibitor blend for the specified time and temperature. After the specified aging, the fixture was removed, cooled to ambient temperatures, the samples removed, and again weighed and measured for hardness. The results of the 160 hour test are set forth in Table 5.

The above test indicated that the second selected polymer material showed stress cracking, which was surprising, because such material should have higher temperature capability. Such cracks appeared along each line sample and seemed to initiate from the line to the o.d. The composite polymer section, formed of 50% of each of the first and second selected polymers appeared not to be homogeneous and each of the selected polymers had formed distinct domains of the separate materials. While the 50/50 blend of the first and second polymers forming the composite polymer material appeared firbrous in nature, it had not split or separated in any way. The hardness change and volume change of the composite material was well within tolerance.

The same test, as above described, was performed, but the time was increased to 504 hours. The results of this test are set forth in table 6.

It should be noted that the method utilised to encapsulate the line with the first and second polymer materials in the above test did not incorporate the pre-heating of the line. The pre-heating step is not necessarily essential, depending upon the percentages of the composite polymer formed from the first and second polymer

materials utilised on the line, and taking into consideration the physical and chemical characteristics which will be found in the environment in which the line is used. When the line is pre-heated, as described above, such cracking is effectively eliminated. A test of an encapsulated line encapsulated as above and tested as above described was tested after 160 hours in the test parameters described above. The results of this test are set forth in Table 7.

The above tests indicate that blends formed from 50% nylon-based polymer and 50% fluoropolymer are not acceptable as encapsulants for lines. Therefore, when practising the present invention, care should be taken to avoid blends with such approximate ranges.

It would be acceptable for the extruded mixture to have a vertically changing composition from substantially 90% of the first-selected polymer and substantially 10% of the second-selected polymer in its upper portions to substantially 90% of the second-selected polymer and substantiaaly 10% of the first-selected polymer in its lowermost portions.

TABLE 1

TYPICAL PROPERTIES OF SELECTED MODIFIED NYLON 6

Property	Value
Specific Gravity	1.08
Yield Tensile Strength, psi	5,300
Ultimate Elongation, %	240
Flexural Strength, psi	4,200
Flexural Modulus, psi	110,000
Notched Izod Impact, ft-lbs/in	6.0
Heat Deflection Temp. @ 264 psi, °F	131
Melting Point, °F	420
Melt Index (Condition Q, gms/10 min)	3-6

TABLE 2

Mechanical Properties	
Tensile strength - at yield, psi	4500
at break, psi	7000
Elongation at break, %	200
Flexural modulus, psi	240,000
Impact resistance, ft-lbs/in	
Izod, notched, 73°F (23°C)	no break
-40°F (-40°C)	2 - 3
Electrical Properties	
Dielectric strength, 0.001 in. thick, V/mil	2000
1/8 in. thick, V/mil	490
Dielectric constant, at 60 Hz	2.6
at 10^3 Hz	2.5
at 10^6 Hz	2.5
Dissipation factor, at 60 Hz	<0.0009
at 10^3 Hz	0.0005
at 10^6 Hz	0.0003
Chemical Resistance, 212°F (100°C)	
Sulfuric acid, 60°Be	no attack
98%	no attack
Nitric acid, concentrated	no attack
Aqua regia	no attack
Sodium hydroxide, 50%	no attack
Flammability	
Oxygen Index, 1/16"	60
UL 94 vertical, 0.007"	94 V-O
Thermal Properties	
Melting Point	240°C (464°F)
Brittleness temperature	<-76°C(-105°F)
Maximum service temperature	150-170°C(300-340°F)
Heat distortion temperature under load (ASTM-D-648) 66 psi stress	115°C (240°F)
264 psi stress	76°C (170°F)
Other Properties	
Radiation resistance	2 x 10^8 rads
Weathering resistance, 3000 hr in weather-ometer	no change in properties
Specific gravity	1.68
Moisture Absorption %	<0.1%
Processing	
Stock temperature	500°-540°F (260-280°C)
Mold (linear) shrinkage, in/in	0.02-0.025

TABLE 3

TENSILE AND FLEXURAL PROPERTIES AT 73°F (23°C)[1]

Property	HALAR® Fluoropolymer
Tensile strength	
at yield, psi	4,500
at break, psi	7,000
Elongation	
at yield, %	5
at break, %	200
Flexural yield strength, psi	7,000
Modulus	
Tensile, psi	240,000
Flexural, psi	240,000

(1) ASTM D 638 and D 790

TABLE 4

Material	Melt Temp. @ Die exit	Melt Press. @ Die psi	Barrel Temperatures, F Rear	Middle	Front	Die
1st Selected Polymer	480-510 F	500-2,000	400 to 550	460 to 530	455 to 530	440 to 530
2nd Selected Polymer	485-560 F	1,000-3,000	410 to 550	450 to 520	480 to 560	480 to 560

TABLE 5

Material	Volume Chg.	Hardness Chg.	Physical Chg.
100% Nylon	+ .2%	+ 5 pts.	None
90% Nylon/10% Halar	+1.4%	- 4 pts.	None
50% Nylon/50% Halar	+2.2%	-10 pts.	None
10% Nylon/ 0% Halar	+1.1%	- 7 pts.	Stress Cracked
100% Halar	+1.2%	- 6 pts.	Stress Cracked

TABLE 6

Material	Volume Chg.	Hardness Chg.	Physical Chg.
100% Nylon	+1.6%	+ 5 pts.	None
90% Nylon/10% Halar	+2.0%	- 8 pts.	Very Minor Crack
50% Nylon/50% Halar	+4.0%	-13 pts.	Extensive Cracking
10% Nylon/90% Halar	+2.6%	- 7 pts.	Extensive Cracking
100% Halar	Not Measurable	Not Measurable	Extensive Cracking, totally split apart

TABLE 7

Material	Volume Chg.	Hardness Chg.	Physical Chg.
100% Nylon	+ .75%	+ 1 pt.	None
90% Nylon/10% Halar	+ .52%	+ 1 pt.	None
50% Nylon/50% Halar	+ 2.1%	- 8 pts.	None
10% Nylon/90% Halar	+ .98%	- 2 pts.	None
100% Halar	+1.18%	- 3 pts.	None

CLAIMS

1. A method of encapsulating an elongate metal line, suitable for positioning in a subsurface well, and comprising extruding a first-selected polymer circumferentially about a first section of the line having a preselected length; extruding a second-selected polymer circumferentially about a second section of the line having a predetermined length; and extruding a composite polymer circumferentially about an intermediate section of the line between the first and second sections, the composition of the composite polymer varying along the intermediate length of the line from a high first-selected polymer/low second-selected polymer composition to a low first-selected polymer/high second-selected polymer composition, such that a continuous extruded encapsulant is formed interconnecting the first- and second-selected polymers, and substantially no part of the extruded mixture having substantially equal proportions of the first- and second-selected polymers.

2. A method according to claim 1, wherein the first-selected polymer is a nylon-based polymer, and the second-selected polymer is a fluoropolymer-based polymer.

3. A method according to claim 1 or claim 2, wherein the composition of the composite polymer varies uniformly with the length of the intermediate section.

4. A method according to any of claims 1 to 3, wherein the metal line is a tubular line for transmitting fluid and/or fluid pressure signals to any portion of the well.

5. A method according to any of claims 1 to 4, wherein the metal line is preheated prior to extruding the composite polymer on the intermediate section.

6. A method according to any of claims 1 to 5, wherein the extruded composite polymer is air or water cooled.

7. A method according to any of claims 1 to 6, wherein the temperature of the composite polymer upon extrusion of the intermediate section is above the melting temperature of the first-selected polymer, but below the decomposition temperature of the first-selected polymer.

8. An encapsulated metal line, suitable for positioning in a hostile subsurface environment, and comprising a first-selected polymer encapsulant circumferentially positioned about a first length of the line; a second-selected polymer encapsulant circumferentially postioned about a second length of the line; and a composite polymer circumferentially positioned about an intermediate section of the line between the first and second lengths, the composition of the composite polymer varying along the intermediate length from a high first-selected polymer/low second-selected polymer composition to a low first-selected polymer/high second-selected polymer composition, such that a continuous encapsulant is formed interconnecting the first- and second-selected polymer encapsulants, and substantially no part of the extruded mixture having substantially equal proportions of the first- and second-selected polymer.

9. A metal line according to claim 8, wherein the first-selected polymer is a nylon-based polymer, and

the second-selected polymer is a fluoropolymer-based polymer.

10. A metal line according to claim 8 or claim 9, wherein the composition of the composite polymer varies uniformly with the length of the intermediate section.

11. A metal line according to any of claims 8 to 10, wherein the metal line is of tubular configuration for transmitting fluid and/or fluid pressure signals through the hostile environment.

12. An encapsulated metal line for use in a subterranean well having a hostile environment in its lower portions, and comprising a first-selected polymer encapsulant circumferentially surrounding first portions of the metal line to be disposed in the upper portion of the well, the first-selected polymer encapsulant comprising a nylon-based polymer; a second-selected polymer encapsulant circumferentially surrounding second portions of the metal line to be disposed in the lower hostile environment portion of the well, the second-selected polymer being fluoropolymer-based polymer; a composite circumferentially surrounding the portions of the metal line between the first and second portions of the metal line and comprising an extruded mixture of the first- and second-selected polymers, the extruded mixture having a vertically changing composition from a substantial majority of the first-selected polymer and a minority of the second-selected polymer in its upper portions to a substantial majority of the second-selected polymer and a minority of the first-selected polymer in it lowermost portions; and substantially no part of the extruded mixture having substantially equal proportions of the first-selected

polymer and the second-selected polymer.

13. An encapsulated line according to claim 12, wherein the extruded mixture has a vertically changing composition from substantially 90% of the first-selected polymer and substantially 10% of the second-selected polymer in its upper portions to substantially 90% of the second-selected polymer and substantially 10% of the first-selected polymer in it lowermost portion.

14. An encapsulated line according to claim 12 or claim 13, wherein the metal line is of tubular configuration for transmitting fluid and or fluid pressure signals through the hostile environment.

15. An encapsulated metal line, substantially as described.

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